## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Commissioner for Patents Washington, D.C. 20231

## DECLARATION OF SHABBIR ATTARWALA

Sir:

- 1. I am employed at Henkel Loctite Corporation, 1001 Trout Brook Crossing, Rocky Hill, CT 06067 ("Loctite"), assignee of the subject application.
- 2. I have been employed at Loctite for 16 years, initially as a Senior Chemist II in the Electronic Product

  Development Group from 1987-1990, then as a Scientist in the

  Cyanoacrylate Product Development Group from 1990-1994, then as a Senior Scientist in the Cyanoacrylate Product Development Group from 1994-1999, and finally from 1999 to the present I became responsible for the management of the product development efforts of the Maintenance and Repair Operation, Machinery Adhesives and Cyanoacrylate Product Development Groups.

- 3. Prior to joining Loctite, I was employed at Kollmorgan Corporation from 1985-1987, as a Manager for Coatings and Adhesives Product Development for Electronic Applications.
- 4. Prior to joining Kollmorgan, I was employed as a research associate and an Adjunct Professor at Polytechnic University, Brooklyn, NY, from where I received my M.S. degree in Organic Chemistry in 1981, may M.S. degree in Polymer Chemistry in 1982 and my Ph.D. degree in Chemistry in 1984. Additionally, I received my B.S. degree with Honors in Chemistry and Physics from Bombay University, Bombay, India in 1973.
- 5. I am a named co-inventor of the invention embodied in the subject application.
- 6. I make this Declaration based on my education and employment experience, which have given me an extensive familiarity of cyanoacrylate adhesives.
- 7. I have reviewed the following documents in making this Declaration:
  - (a) U.S. Patent Application No. 10/078,0056 ("the subject application");
  - (b) Preliminary Amendment dated March 12, 2002
     ("the Preliminary Amendment");
  - (c) Office Action mailed August 2, 2002 issued in the subject application ("the Office Action");
  - (d) Mikune, U.S. Patent No. 5,824,180 ("the '180
     patent");
  - (e) Mikune, European Patent Document 769 721
     ("the EP '721 patent publication");

- (f) Attarwala, U.S. Patent No. 5,328,944 ("the '944 patent");
- (g) Gatechair, U.S. Patent No. 4,707,432 ("the '432 patent"); and
- (h) H.V. Coover, D.W. Dreifus and J.T. O'Connor, "Cyanoacrylate Adhesives" in <u>Handbook of Adhesives</u>, 27, 463-77, I. Skeist, ed., Van Nostrand Reinhold, New York, 3rd ed. (1990) ("Coover");
- 8. The invention as presently defined by Claim 1 of the subject application is directed to a composition, which includes:
- (a) a 2-cyanoacrylate monomer of the formula  $H_2C=C(CN)-COOR$ , wherein R is selected from the group consisting of  $C_{1-15}$  alkyl, alkoxyalkyl, cycloalkyl, alkenyl, aralkyl, aryl, allyl and haloalkyl groups,
  - (b) a metallocene component,
- (c) a polymerisingly effective amount of a photoinitiator component other than the metallocene component to render the composition capable of photocuring in air upon exposure to at least one type of electromagnetic radiation selected from the group consisting of ultraviolet light, visible light, electron beam, x-ray and infrared radiation, and
- (d) one or more sulfur-containing compounds selected from the group consisting of sulfonates, sulfinates, sulfates, and sulfites.
  - 9. The inventive photocurable cyanoacrylate

composition is a sensitive system, even more so than a conventional cyanoacrylate composition, in that the addition of agents to perform certain functions may have adverse effects on the stability, for instance, which may be dramatic.

- 10. The Office Action has continued to reject Claims 1, 4-7, 11-14, 16-22, 26-34 and 36 under 35 U.S.C. § 103(a) as being allegedly obvious over the '180 patent or the EP '721 publication (pages 6-9), and under Section 103(a) as being allegedly obvious over the EP '721 publication, in view of the '944 patent and further in view of the '432 patent (pages 9-10). The Office Action further continues to reject Claims 23-26 and 30-32 under Section 103(a) as being allegedly obvious over the '180 patent or the EP '721 publication, in view of the '944 patent and further in view of Coover (page 10).
- 11. The '180 patent is directed to and claims a method of bonding an artificial nail to a nail with an adhesive or of bonding a decorative article to an artificial nail or a nail using an adhesive. In the '180 patent, the adhesive is reported as a photocurable cyanoacrylate adhesive, which includes an  $\alpha$ -cyanoacrylate and a metallocene compound of a transition metal of Group VIII of the periodic table and aromatic electron system ligands. The photocurable cyanoacrylate adhesive may also include a cleavage photoinitiator, specific ones of which being reported.
  - 12. The EP '721 publication claims a photocurable

composition comprising (a) a 2-cyanoacrylate, (b) a metallocene and (c) an acyl cleavage photoinitiator. The metallocene compound (b) is represented by specified metal dicyclopentadienyl complexes with the transition metal of group VIII of the periodic table comprises iron, osmium, ruthenium, cobalt or nickel. The EP '721 publication makes passing reference to the addition of optional ingredients including anionic-polymerization inhibitors; radical-polymerization inhibitors; thickening agents; specific curing accelerators; plasticizers; tougheners; and heat stabilizers; perfumes; dyes; and pigments.

- described in the Specification and speaks to cyanoacrylate monomer adhesive formulations that include sulfur containing compounds to improve the thermal resistance of the cured adhesive formulation. No express disclosure, teaching or suggestion is present in the '944 patent, as it relates to photocuring of cyanoacrylates, or the use of sulfur containing compounds to improve shelf life stability.
- 14. The '432 patent is described in the Specification, and speaks to a free radical polymerizable composition which includes (a) polymerizable partial esters of epoxy resins and acrylic and/or methacrylic, and partial esters of polyols and acrylic acid and/or methacrylic acid, and (b) a photoinitiator blend of a cyclopentadienyl iron complex and a sensitizer or photoinitiator, such as an acetophenone.

- obviousness formula advanced in the Office Action. While one of the chemicals to which the '432 patent refers is in part an acrylate, cyanoacrylates which are used in the compositions which are defined by the subject application, have quite disparate properties and have developed as a separate class of chemicals, particularly in the adhesives industry. Thus, the acrylate- and epoxy-containing compositions of the '432 patent are not the cyanoacrylates used in the present invention. And there would be no reason to look to these chemicals with the expectation that cynanoacrylates would behave in a similar manner, let alone a cyanoacrylate curable by exposure to electromagnetic radiation alone.
- which they are described as quick-setting materials which cure to clear, hard glassy resins, useful as sealants, coatings, and particularly adhesives for bonding together a variety of substrates. Coover does not teach or suggest the ability of curing cyanoacrylates through exposure to radiation in the visible range of the electromagnetic spectrum. Thus, Coover adds nothing to the obviousness formula advanced in the Office Action.
- 17. Accordingly, based on my employment experience, formal education and review of the documents identified in paragraph 7, I do not see that a person of ordinary skill in the art, such as myself, would be motivated by the '180 patent or the

EP '721 publication, or for that matter from any of the cited documents of record to which reference is made in paragraph 7, to use certain sulfur containing compounds to formulate a photocurable cyanoacrylate composition as Applicants have done.

- 18. Moreover, under my guidance, experiments were conducted in which the affect of the addition of ethylene sulfite (one of the expressly recited sulfur containing compounds) on ethyl-2-cyanoacrylate and on a photocurable cyanoacrylate composition comprising ethyl-2-cyanoacrylate in combination with ferrocene as a metallocene and "IRGACURE" 819 [bis(2,4,6-trimethyl benzoyl) phenyl phosphine oxide] as a photinitiator was studied. As a result of the study it was determined that the addition of ethylene sulfite to the photocurable cyanoacrylate composition improved the shelf life stability significantly (measured as a function of the increase of viscosity at room temperature over time).
- 19. More specifically, experiments were conducted in which formulations of 10% PMMA-thickened ethyl-2-cyanoacrylate (Sample A), 10% PMMA-thickened ethyl-2-cyanoacrylate together with 0.005% ferrocene and 0.25% "IRGACURE" 819 (Sample B), and 10% PMMA-thickened ethyl-2-cyanoacrylate together with 0.005% ferrocene and 0.25% "IRGACURE" 819 and 0.05% ethylene sulfite (Sample C) were prepared and evaluated for shelf life stability using an accelerated heat aging technique. In these experiments, Samples A-C were placed in glass tubes in a temperature-

controlled oven at 82°C, and the number of days to gellation was measured. Sample A was observed to gel to a state of non-flowability after 25 days at that temperature condition; Sample B gelled after 7 days; and Sample C gelled after 10 days.

- 20. While performing not as well as Sample A (without the photoinitiating system or the ethylene sulfite), Sample C (with ethylene sulfite) demonstrated a nearly 43% increase in stability as compared to Sample B (without the ethylene sulfite).
- 21. In addition, experiments were conducted in which formulations of 9.8% PMMA-thickened ethyl-2-cyanoacrylate (Sample D), 9.8% PMMA-thickened ethyl-2-cyanoacrylate together with 0.01% ferrocene and 0.5% "IRGACURE" 819 (Sample E), and 9.8% PMMA-thickened ethyl-2-cyanoacrylate together with 0.01% ferrocene and 0.5% "IRGACURE" 819 and 0.025% ethylene sulfite (Sample F) were prepared and evaluated as above. In these experiments, Sample D gelled after 18 days at that temperature condition; Sample E gelled after 3 days; and Sample D gelled after 6 days.
- 22. While performing not as well as Sample D (without the photoinitiating system or the ethylene sulfite), Sample F (with ethylene sulfite) demonstrated a 50% increase in stability as compared to Sample E (without the ethylene sulfite).
- 23. Accordingly, based on my employment experience and formal education, I believe that none of the cited documents of record disclose, teach or suggest the improvements observed above, and I believe that a person of ordinary skill in the art,

such as myself, would not have expected to achieve that improvement based on such documents.

24. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

March 15, 2003 Date

Shabbir Attarwala